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WATER ABSORPTION AND DESORPTION IN SHUTTLE ABLATOR AND
INSULATION MATERIALS

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16. ABSTRACT Shuttle systems ablator and insulation materials have undergone water soak with subsequent water desorption in vacuum. Water accumulation in these materials after a soak for 24 hours ranged from +1.1% for orbiter tile to +161% for SRB MSA-1. After 1 minute in vacuum, water retention ranged from none in the orbiter tile to +70% for SRB cork.			
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TECHNICAL MEMORANDUM

WATER ABSORPTION AND DESORPTION IN SHUTTLE ABLATOR AND INSULATION MATERIALS

I. INTRODUCTION

Some concern exists as to the amount of moisture that ablator and insulation materials on the Shuttle Orbiter, External Tank and Solid Rocket Booster can absorb during extended periods on the launch pad. Any appreciable absorbed water retained in these systems would add weight which would be reflected in reduced thrust during the nine minutes of launch. This investigation was undertaken to obtain a relative measure of the water accumulation of these materials during water soak and their subsequent water retention/desorption properties during vacuum evaporation. Data generated here would be used in an assessment of the probability of appreciable water accumulation in these Shuttle systems.

Two sets of test samples consisting of Orbiter tile, SRB ablators, MSA-1 and Cork, and External Tank ablator, SLA-561, and Insulator, 488 Foam, were evaluated. A set of these materials in a large volume/surface configuration (large specimens) shown in Figure 1 was allowed to soak in water for 24 hours then subjected to vacuum evaporation. Materials were sized in 2.5 cm (1 inch) cubed specimens for the tile, 488 foam and SLA-561 and in (2.5 x 2.5 x .6) cm volume specimens for the cork and MSA-1. System pressure profiles and RGA water peak data versus time were generated for these samples to provide comparative data. The second set in a small volume/surface configuration (small specimens) was allowed to soak in water until saturation or an absorption limit was achieved then subjected to vacuum where its weight was monitored during desorption. These samples, in providing comparative data, were sized to furnish specific insitu weight loss information versus time and pressure.

II. TEST EQUIPMENT

The vacuum system, Cahn Microbalance, Residual Gas Analyzer (RGA) and associated instrumentation shown in Figure 2 were utilized to evaluate these materials. The vacuum system is an all stainless steel high vacuum system with metal seals which is pumped with a 4 inch diffusion pump that is backed by a 500 liters/minute roughing pump and trapped with a LN₂ baffle and cryopump. This system has a pumping speed of 500 liters/second and a volume of approximately 0.5 ft³. This configuration was used to desorb the test specimens with the roughing pump until the chamber pressure was sufficiently low for diffusion pump operation.

A UTI 100C-12 residual gas analyzer with a sensitivity of 300 amperes/torr (emission current 2 milliamperes) was used to monitor desorption of water in the large volume/surface test specimens. When the system pressure was 5×10^{-6} torr (maximum RGA operating pressure) or less the RGA was activated and the water peak, 18 AMU, was monitored.

An insitu weight measurement of water desorption in the small volume/surface specimens was made using a Cahn RG electrobalance with a sensitivity of 0.1 microgram and a test setup resolution of 25 micrograms. The weight of the test specimen with absorbed water was limited to 100 milligrams. Weights of the specimens taken during water soak and after vacuum desorption were made on a Mettler analytical balance.

III. DATA PRESENTATION

Table I shows the large volume/surface specimen weights in the conditions of: dry as received, after water soak for 24 hours, prior to vacuum exposure, and immediately following vacuum desorption. Prior to vacuum exposure weights were, in some cases, determined just after soak while others were determined several hours later depending on the test readiness of the vacuum system and associated equipment. It can be seen that some evaporation occurred during the wait for the test chamber. Vacuum exposure for these samples varied from one to six hours. The variation in vacuum exposure times resulted from the ease/difficulty of vacuum desorption of the water soaked materials.

Figures 3 through 7 are plots of the system pressure and water peak height versus time for the large samples. RGA water peak height data could be generated only when the system pressure was well within the 10^{-6} torr range. It could not be acquired within a reasonable test time for the MSA-1.

Table II contains the small volume/surface specimen weights in the conditions of: dry as received, after water soak to saturation, prior to vacuum exposure, and immediately following vacuum desorption. Dry weights ranged from 25 to 50 milligrams and were sized so that their water soaked weight would not exceed the Cahn balance test configuration limit of 100 milligrams. Nominal soak to saturation time ranged from 18.5 to 22 hours. Saturation for some of the samples occurred at less than 18.5 hours but was undetected since it occurred during overnight soak.

Figure 8 is a plot of orbiter tile specimen weight versus time as the system pressure was reduced. Figure 9 indicated the rate of weight change in the tile as it underwent water soak. Also shown on the graph is a plot of the increase in weight

of the sample in air versus time beginning immediately after the completion of the vacuum desorption test. The other materials are treated similarly in Figures 10 through 17.

IV. SUMMARY AND CONCLUSIONS

Table III summarizes the water absorption/desorption characteristics of the materials described here. Further analyses of these materials are provided in the following paragraphs.

A. Orbiter Tile

The orbiter tile does not readily absorb water. The large specimen, which absorbed about 1% water in a 24 hour water soak and evaporated almost to its original weight during the test delay, vacuum desorbed in about 30 minutes. The water peak height shown along with the pressure profile in Figure 3 dropped by a factor of 2 in 13 minutes in the pressure range from 3×10^{-6} to 0.3×10^{-6} torr. The small specimen which was allowed to soak to saturation or to an absorption limit was weighed during the water soak and ceased to absorb after reaching an increase in weight of +14% in about 17 hours (Figure 9). Although this specimen has a small volume/surface ratio it is suspected that most of the 14% (water) is on or near the specimen surface. Supporting evidence is provided in Figure 8 where approximately 40% of the 14% (water) had evaporated just prior to the vacuum initiation. The specimen reached its dry weight in less than 1 minute in the 20 to 100 micron pressure range. The specimen desorbed to a weight below its dry weight and did not regain any of this weight after about 1.3 hours in air (Figure 9). This would suggest the material outgassed products other than water and/or the difficulty of water being absorbed back into the material in the ambient humidity (35-45%) of the laboratory. Since little evidence of appreciable outgassed products is observed in the pressure profile of Figure 3 it appears reasonable to assume the weight loss below the specimen dry weight is attributable mostly to water taken on by the material during long exposures in a moderately humid environment.

B. 488 Foam

The 488 foam easily absorbs water in a saturated environment and desorbs in a vacuum environment; however, it does not readily absorb from the ambient humidity (35-45%) in the laboratory environment. The large specimen increased in weight by +40% during the 24 hours of water soak but evaporated to +6% during the 1.5 hours wait prior to testing. The water peak shown with the pressure profile in Figure 4

dropped by a factor of 2.5 in 30 minutes during vacuum desorption. The small specimen treated similarly to the tile increased in weight by +224% during the soak of about 21.5 hours with most of the increase occurring within the first 5 hours of soak (Figure 11). This specimen vacuum desorbed to its dry weight in about 15 minutes (Figure 10). No weight increase was detected in this material during 3.5 hours in the ambient humidity of the laboratory. Since the dry weight and the after vacuum weight are essentially the same, this material apparently does not contain potential outgassing products.

C. SLA-561

The SLA-561 easily absorbs water from a saturated environment and from the ambient humidity (35-45%) in the laboratory. It vacuum desorbs at a moderate rate when compared to the tile and 488 foam. The large specimen increased in weight by +22% during the 24 hours of water soak. The pressure profile (Figure 5) in deviating from the exponential shows pressure sensitive outgas products. Since the pressure remained high in the system for about 2 hours little information was gained from the RGA data.

The small specimen weight increase was 62% during the 18.5 hours of water soak; it vacuum desorbed to 80% of its dry weight in 14 minutes (Figure 12). The pressure profile (Figure 5) and the slight weight gain in air after vacuum desorption (Figure 13) indicate that the -2.2% weight loss below dry weight are due to a loss of both water and additional outgassed products.

D. Cork

The cork rapidly absorbs water in both air and in a saturated environment and is very difficult to vacuum desorb. The large specimen gained weight during the 24 hour soak but evaporated to +15% in the 19 hours wait prior to desorption. The system pressure desorption curve taken over 5 hours (Figure 6) shows evidence of additional outgassed products. The small specimen showed a water pickup of +126% in 20 hours and vacuum desorbed to less than half its saturated weight in 15 minutes. It began to pick up water in air immediately following vacuum desorption.

E. MSA-1

The MSA-1 readily takes up water in a saturated environment but does not absorb in the laboratory environment (35-45% humidity) after vacuum desorption. When water saturated, its vacuum desorption characteristics were similar to the cork. The pressure profile (Figure 7) of the large specimen shows two regions of vapor pressure sensitive products. A white deposit on the edges of the material after

vacuum desorption also confirmed the loss of components other than water. No RGA water peak height data were available for this material. Small specimen data (Figure 17) show the MSA-1 to immediately take on water in a saturated environment; it accumulated a +217% weight increase in 22 hours of water soak. It vacuum desorbed to about 42% of its dry weight in 15 minutes.

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Orbiter Tile

MSA-1



488 Foam

SLA-561



Cork

FIGURE 1. SHUTTLE SYSTEMS ABLATOR AND INSULATION MATERIALS



FIGURE 2. VACUUM SYSTEM AND ASSOCIATED TEST EQUIPMENT

**TABLE I. WATER DESORPTION DATA FOR LARGE VOLUME/SURFACE
SHUTTLE MATERIAL TEST SPECIMENS**

Orbiter Tile (2.5 x 2.5 x 2.5) cm

<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	2.3002	—
Water Soaked (24 Hrs)	2.3256	+1.1
Prior to Vacuum (2.5 Hrs)	2.2955	~0
After Vacuum Exp (1 Hr)	2.2950	~0

488 Foam (2.6 x 2.6 x 2.6) cm

<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	.6354	—
Water Soaked (24 Hrs)	.8915	+40
Prior to Vacuum (1.5 Hrs)	.6735	+6
After Vacuum Exp (1 Hr)	.6279	-1.1

SLA-561 (2.6 x 2.6 x 2.6) cm

<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	4.0706	—
Water Soaked (24 Hrs)	4.9686	+22
Prior to Vacuum (0 Hr)	4.9686	+22
After Vacuum Exp (2.5 Hrs)	4.0704	~0

Cork (2.6 x 2.6 x .6) cm

<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	1.7937	—
Water Soaked (24 Hrs)	2.8646	+60
Prior to Vacuum (19 Hrs)	2.0644	+15
After Vacuum Exp (5 Hrs)	1.6816	-6

MSA-1 (2.6 x 2.6 x .7) cm

<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	1.1181	—
Water Soaked (24 Hrs)	2.9177	+161
Prior to Vacuum (0 Hr)	2.9177	+161
After Vacuum Exp (6 Hrs)*	1.0913	-2.4

* White substance on edges of sample, showing evidence of losing components other than H₂O. Also a change in the sample's color was detected.

TABLE II. WATER DESORPTION DATA FOR SMALL VOLUME/SURFACE
SHUTTLE MATERIAL TEST SPECIMENS

<u>Orbiter Tile (1.25 x .55 x .45) cm</u>		
<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	.05000	—
Water Soaked (18.5 Hrs)	.05700	+14
Prior to Vacuum (0 Hr)	.05650	+12
After Vacuum Exp (1 Hr)	.04825	-3.5
<u>488 Foam (2.1 x .65 x .5) cm</u>		
<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	.02500	—
Water Soaked (2.5 Hrs)	.08100	+224
Prior to Vacuum (0 Hr)	.08045	+222
After Vacuum Exp (1 Hr)	.02495	-.2
<u>SLA-561 (.95 x .45 x .45) cm</u>		
<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	.04050	—
Water Soaked (18.5 Hrs)	.06550	+62
Prior to Vacuum (0 Hr)	.06550	+62
After Vacuum Exp (1 Hr)	.03960	-2.2
<u>Cork (.45 x .4 x .3) cm</u>		
<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	.03000	—
Water Soaked (20 Hrs)	.06770	+126
Prior to Vacuum (0 Hr)	.06770	+126
After Vacuum Exp (2.5 Hrs)	.04775	+59
<u>MSA-1 (.7 x .4 x .35) cm</u>		
<u>Specimen Condition</u>	<u>Weight, Grams</u>	<u>Percentage Change</u>
Dry, as Received	.02500	—
Water Soaked (22 Hrs)	.07935	+217
Prior to Vacuum (0 Hr)	.07935	+217
After Vacuum Exp (2 Hrs)	.02412	-3.5

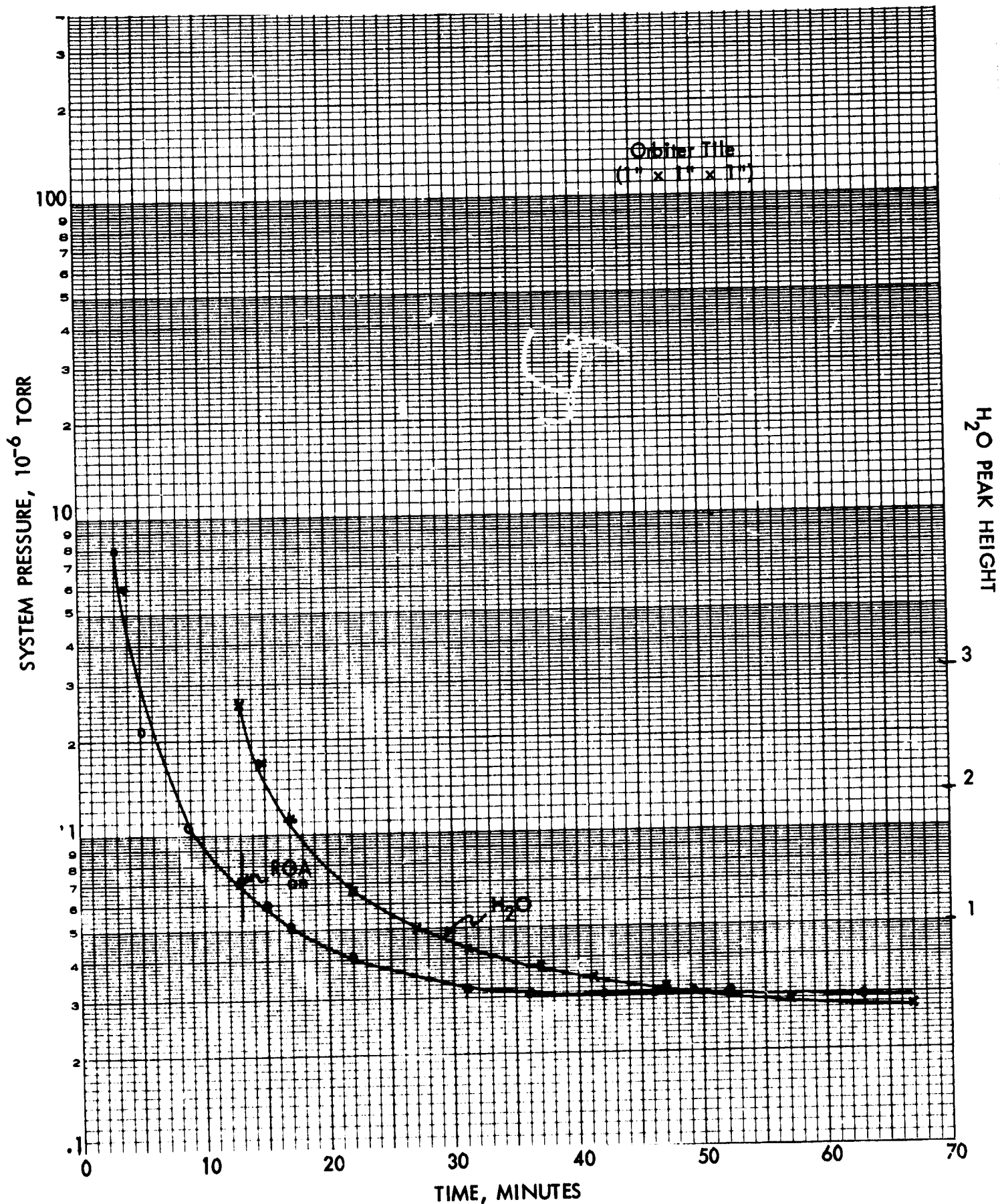


FIGURE 3. SYSTEM PRESSURE AND H₂O PEAK DURING VACUUM DESORPTION OF ORBITER TILE

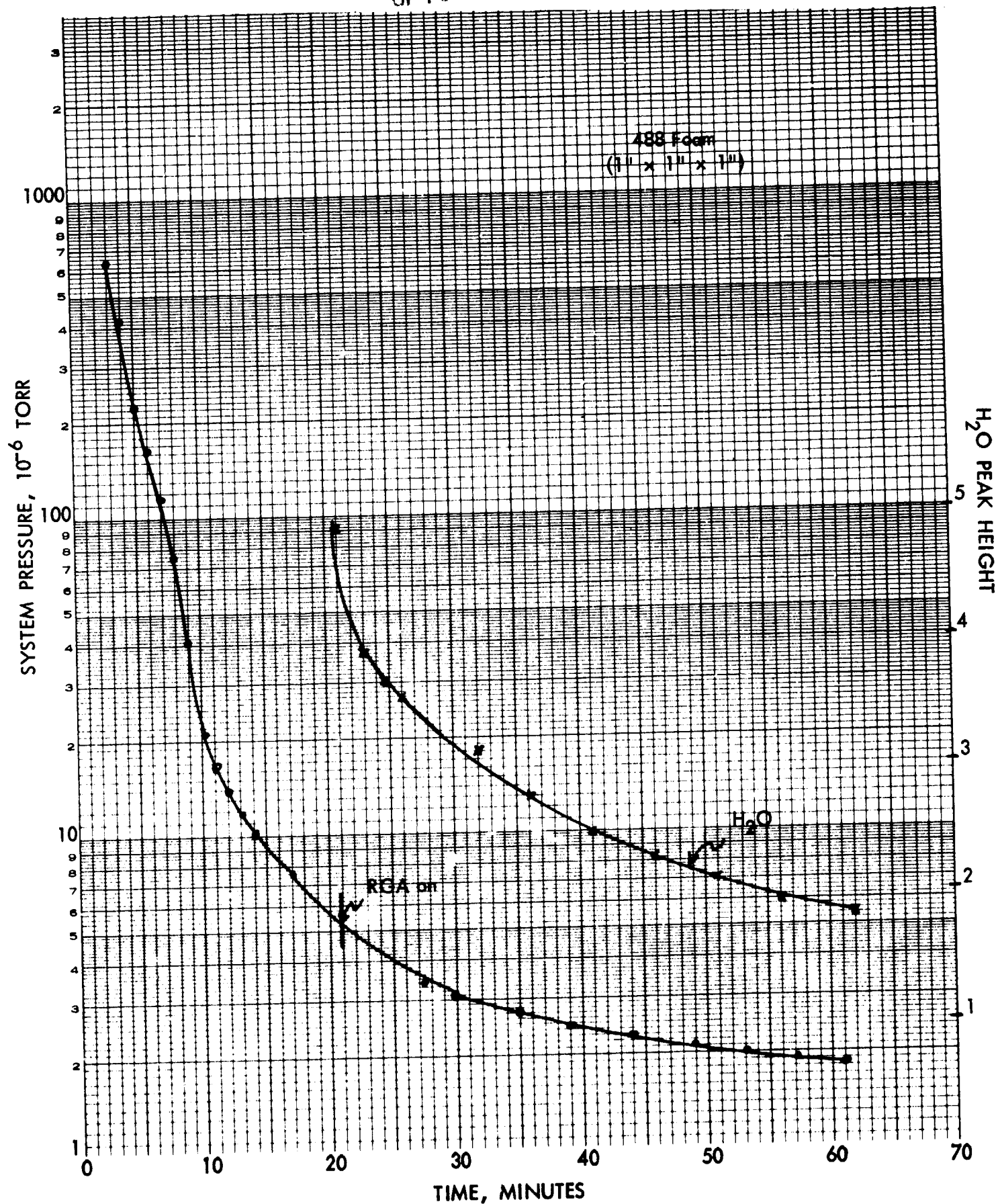


FIGURE 4. SYSTEM PRESSURE AND H₂O PEAK DURING VACUUM DESORPTION OF 488 FOAM

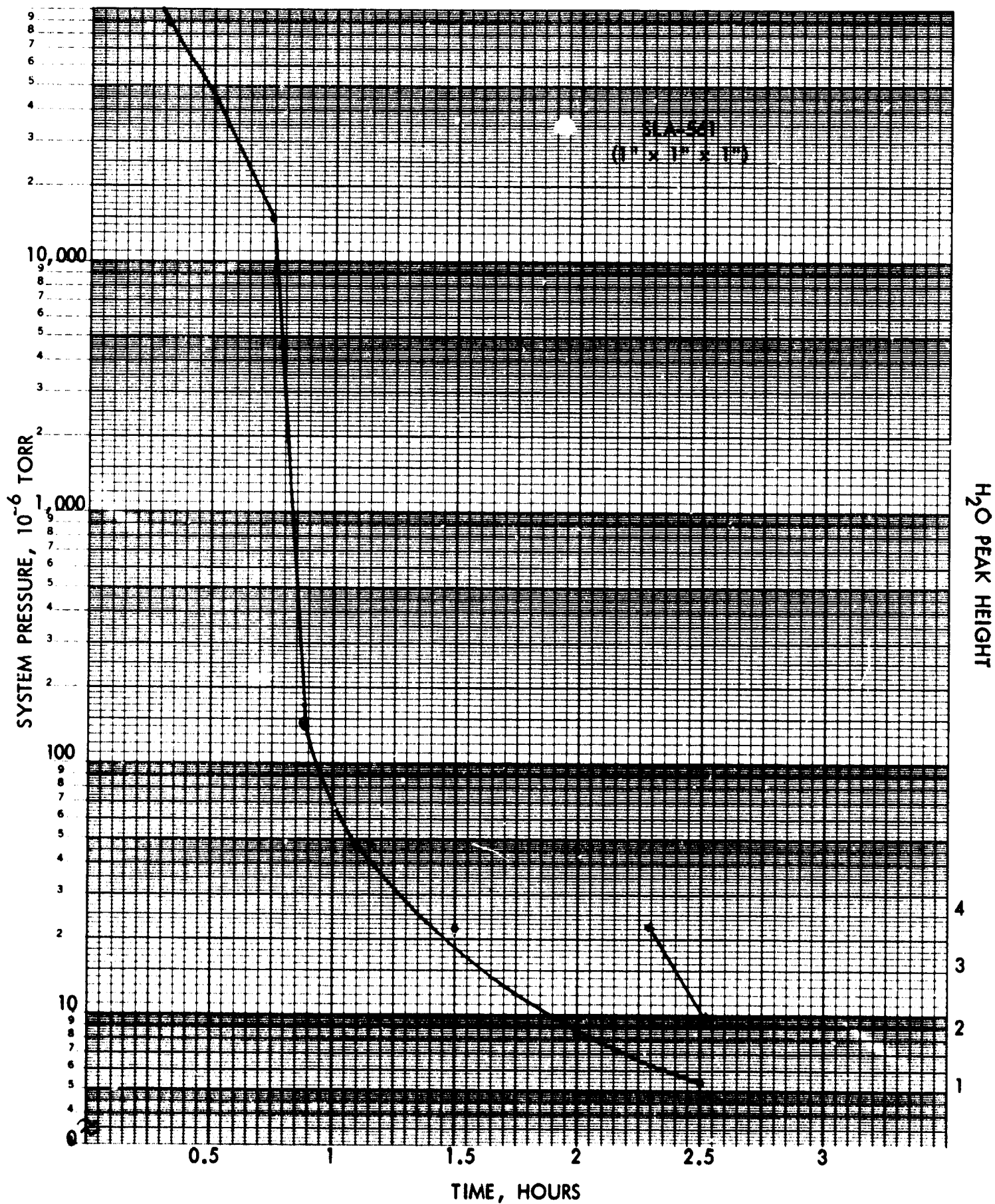


FIGURE 5. SYSTEM PRESSURE AND H₂O PEAK DURING VACUUM DESORPTION OF SLA-561

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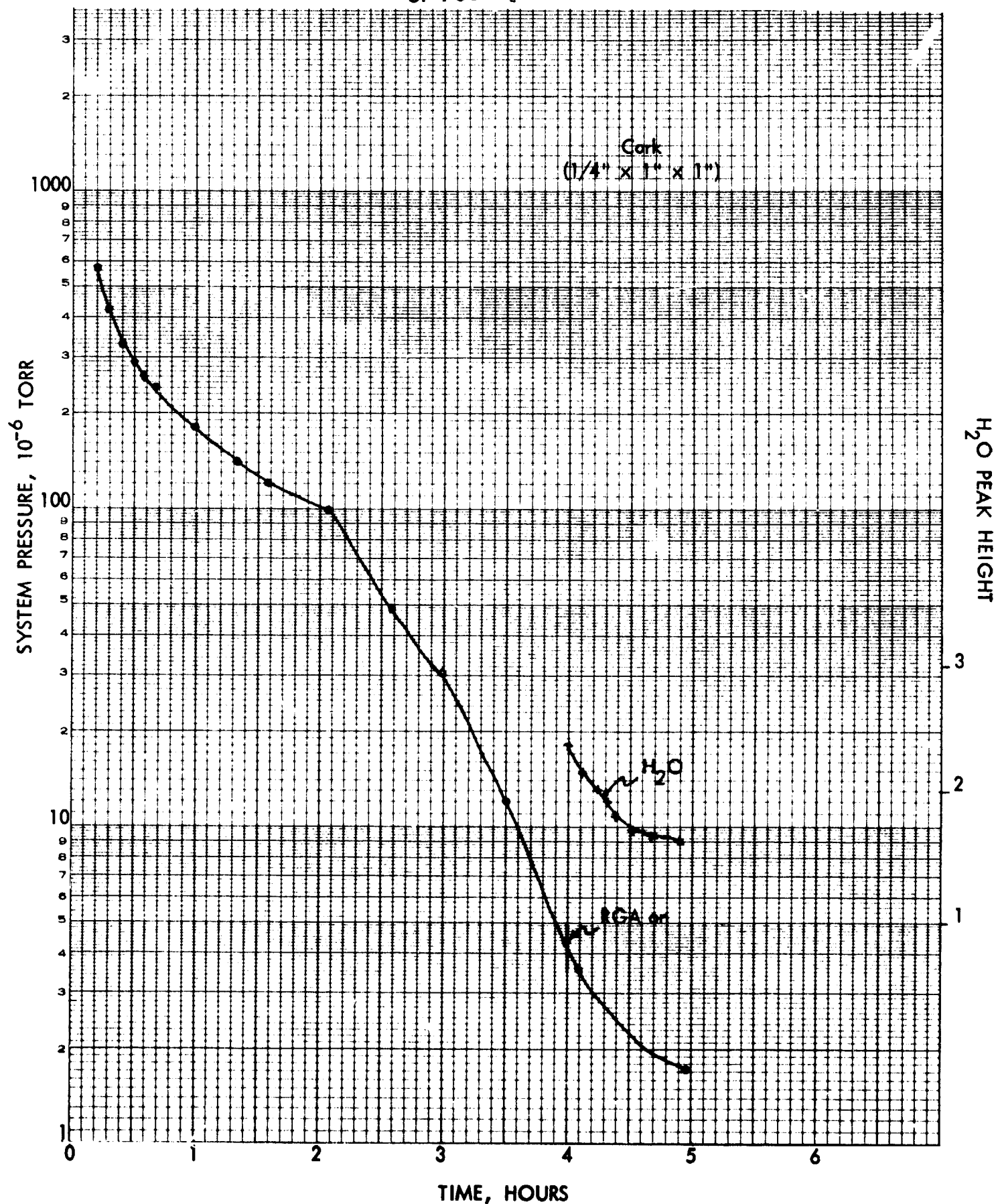


FIGURE 6. SYSTEM PRESSURE AND H_2O RGA PEAK DURING VACUUM DESORPTION OF CORK

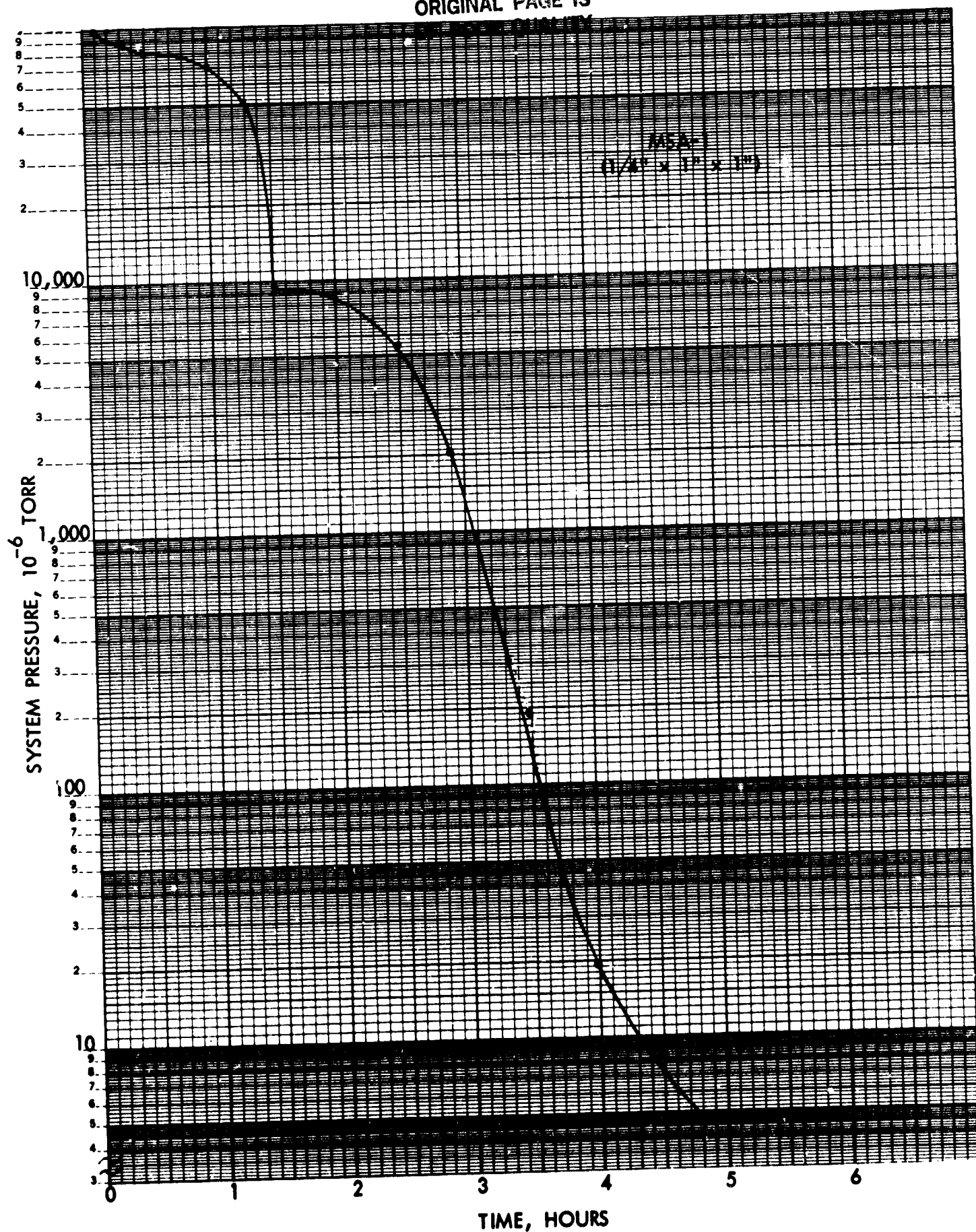


FIGURE 7. SYSTEM PRESSURE DURING VACUUM DESORPTION OF MSA-1

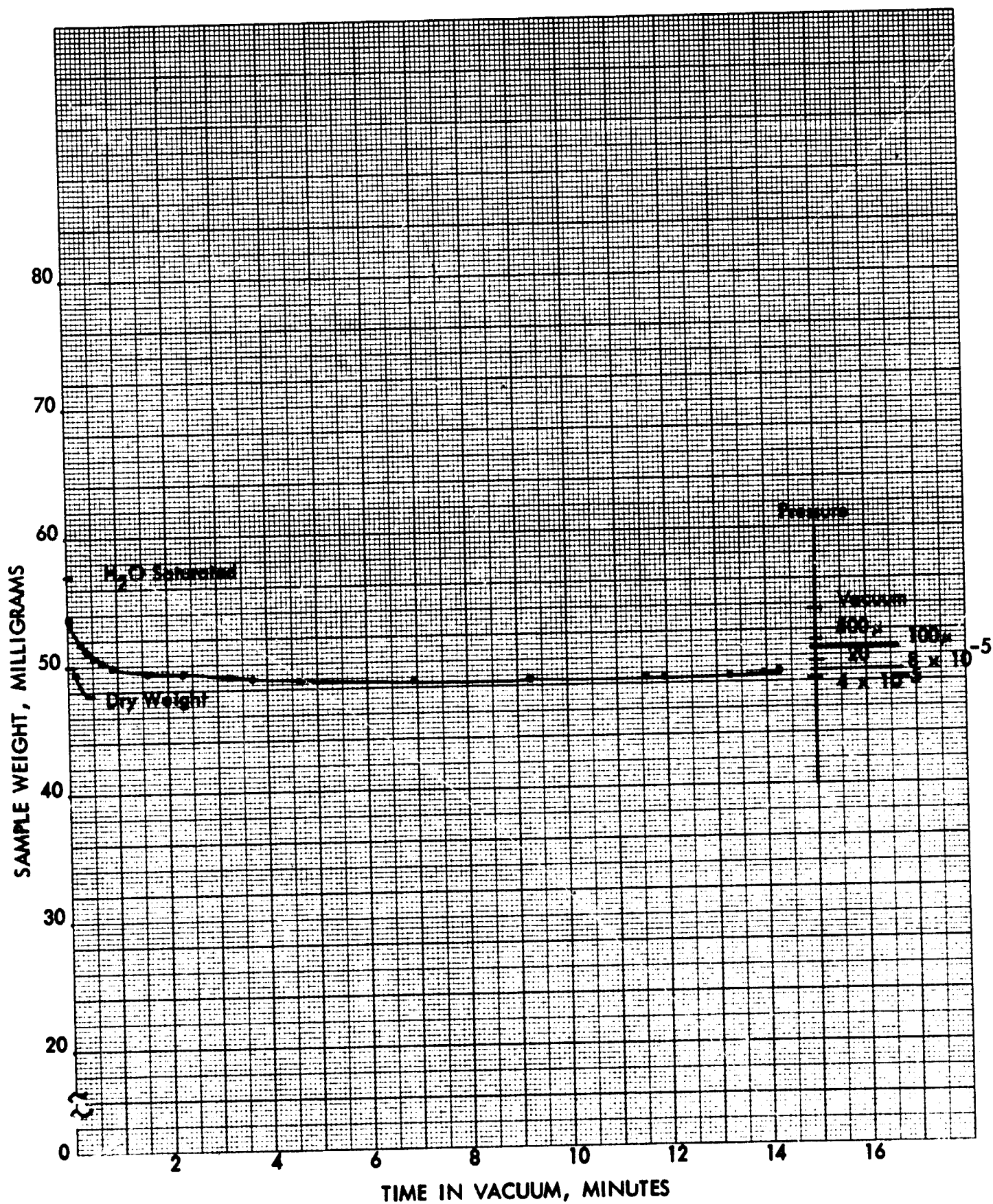


FIGURE 8. WATER DESORPTION OF ORBITER TILE IN VACUUM

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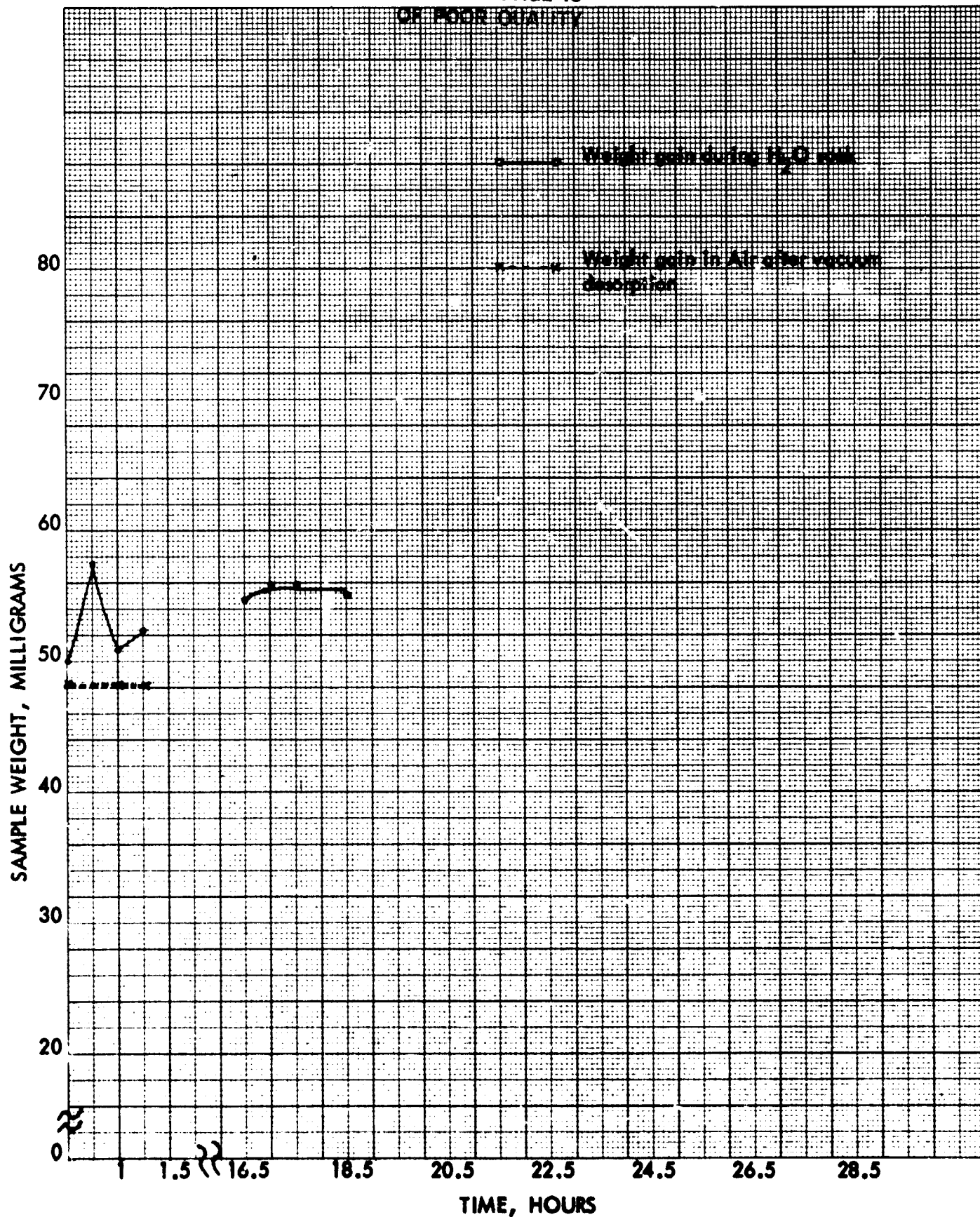


FIGURE 9. WEIGHT GAIN OF ORBITER TILE DURING H₂O SOAK AND IN AIR AFTER VACUUM DESORPTION

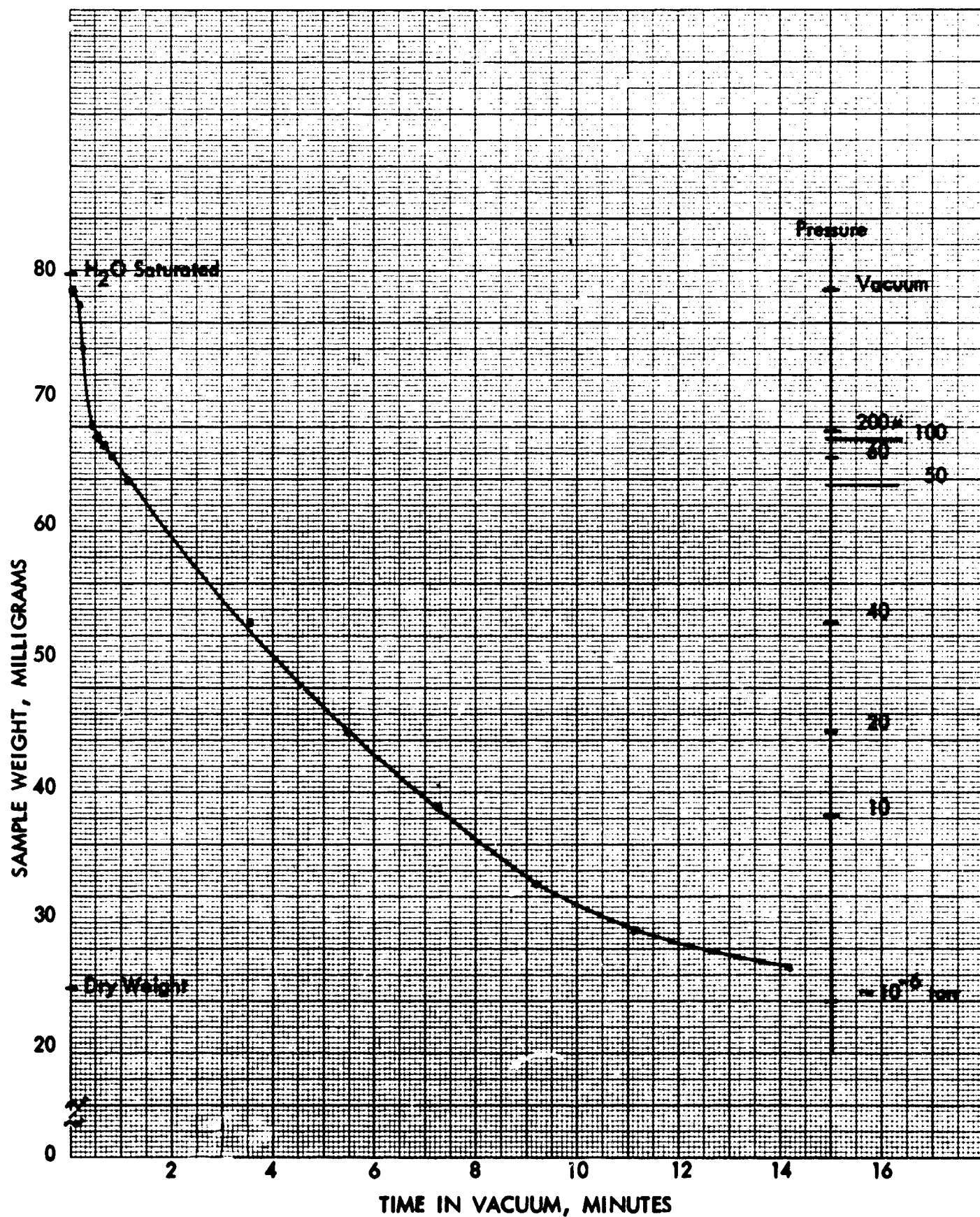


FIGURE 10. WATER DESORPTION OF 488 FOAM IN VACUUM

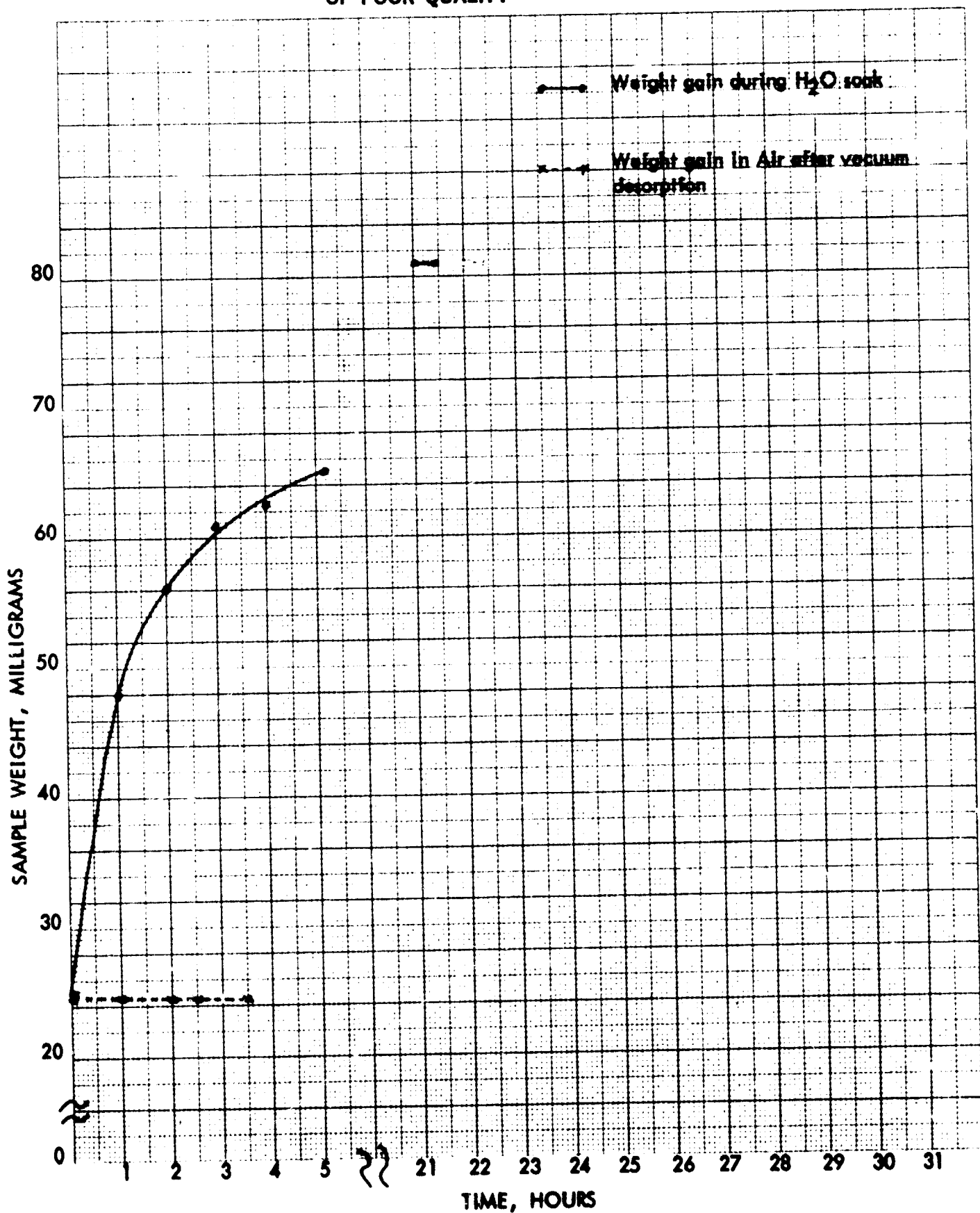


FIGURE 11. WEIGHT GAIN OF 488 FOAM DURING H₂O SOAK AND IN AIR AFTER VACUUM DESORPTION

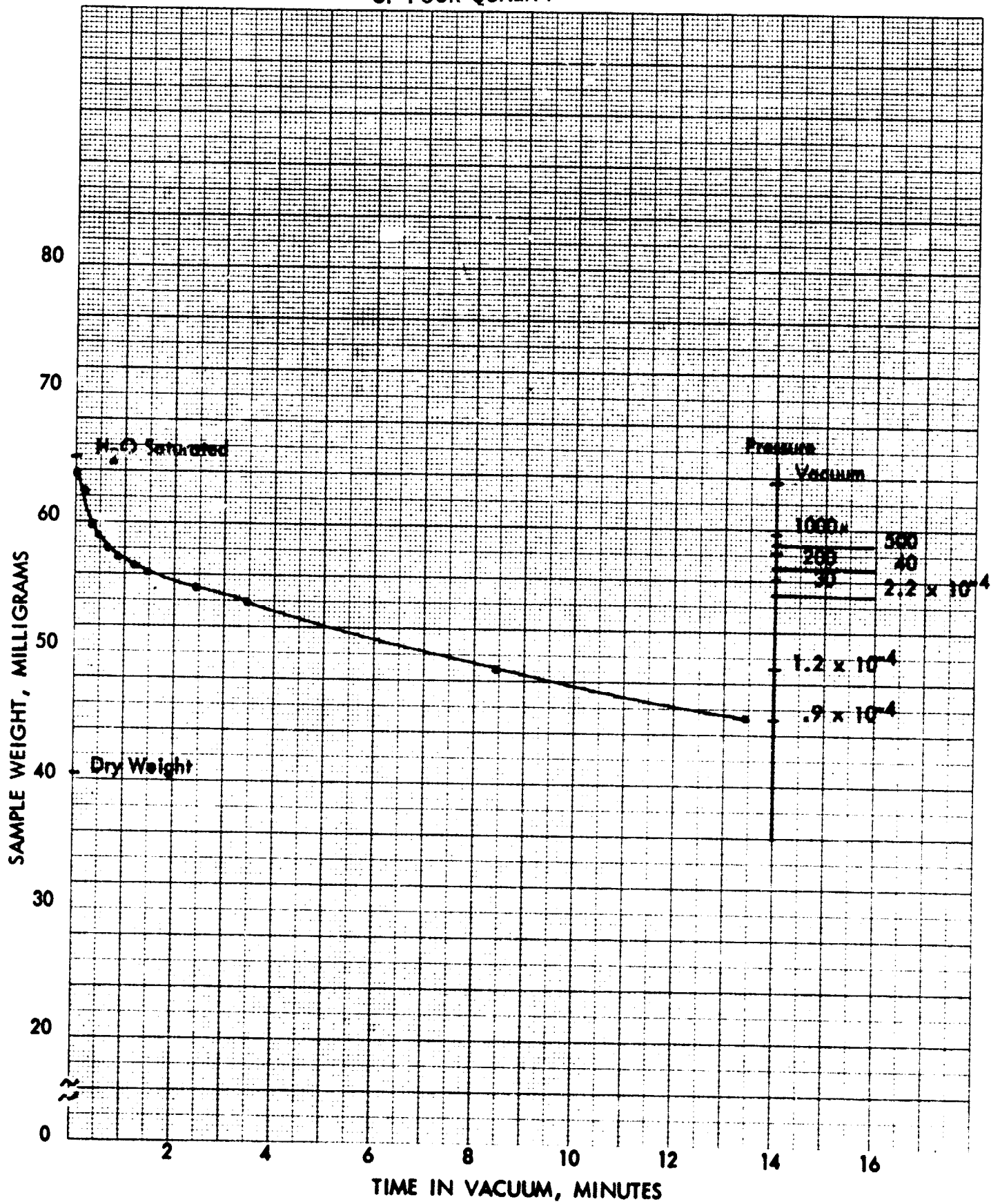


FIGURE 12. WATER DESORPTION OF SLA-561 IN VACUUM

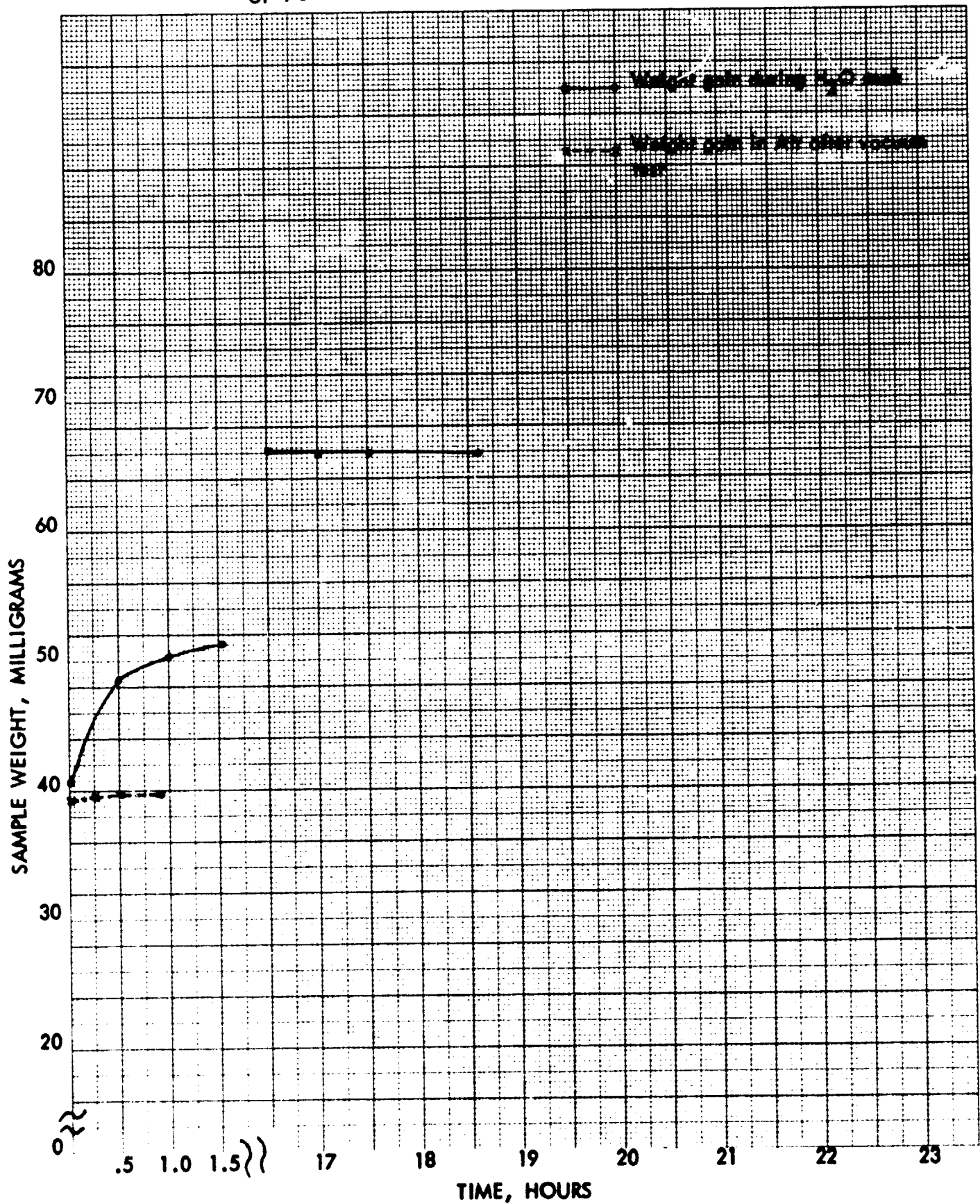


FIGURE 13. WEIGHT GAIN OF SLA-561 DURING H₂O SOAK AND IN AIR AFTER VACUUM DESORPTION

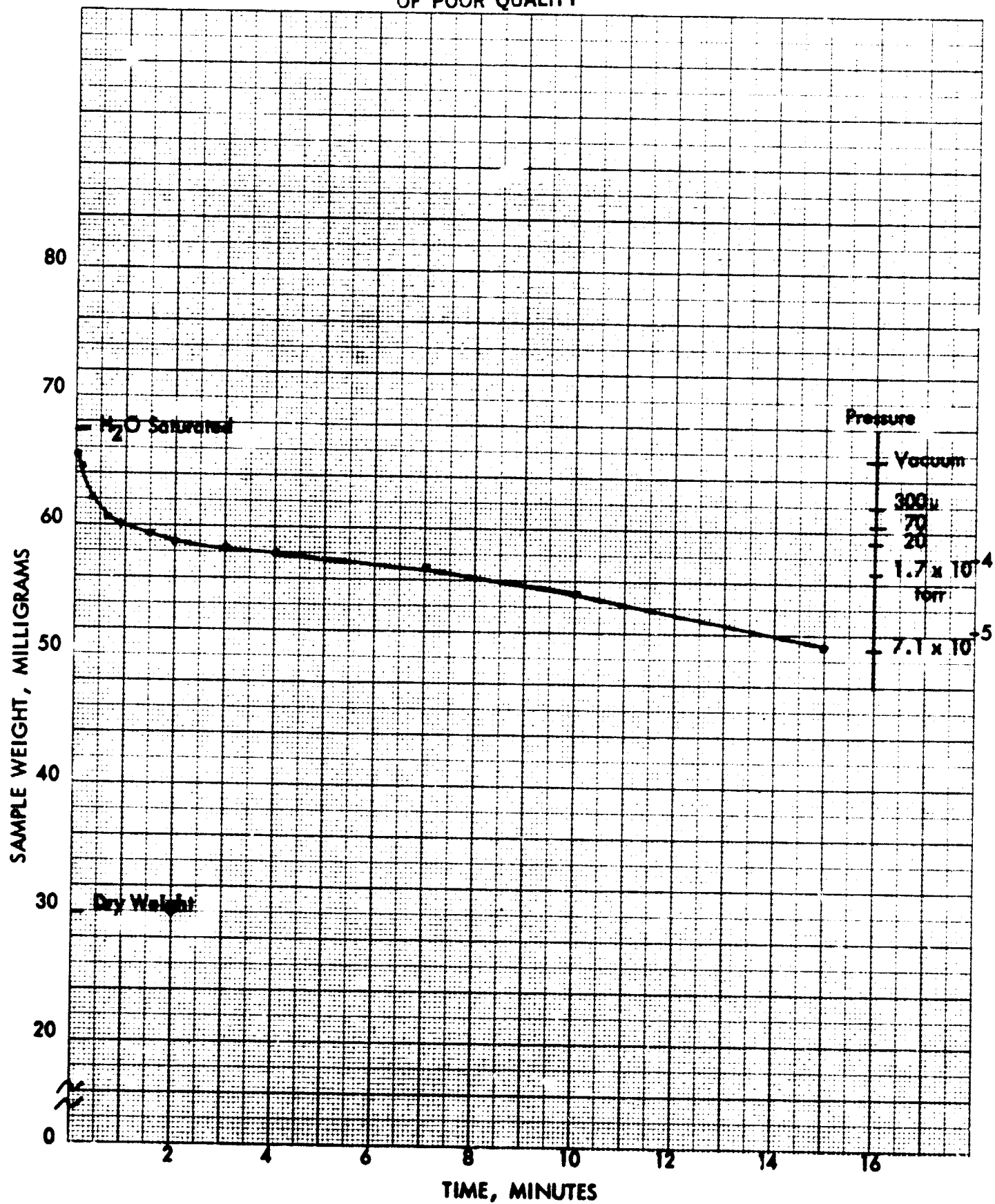


FIGURE 14. WATER DESORPTION OF CORK IN VACUUM

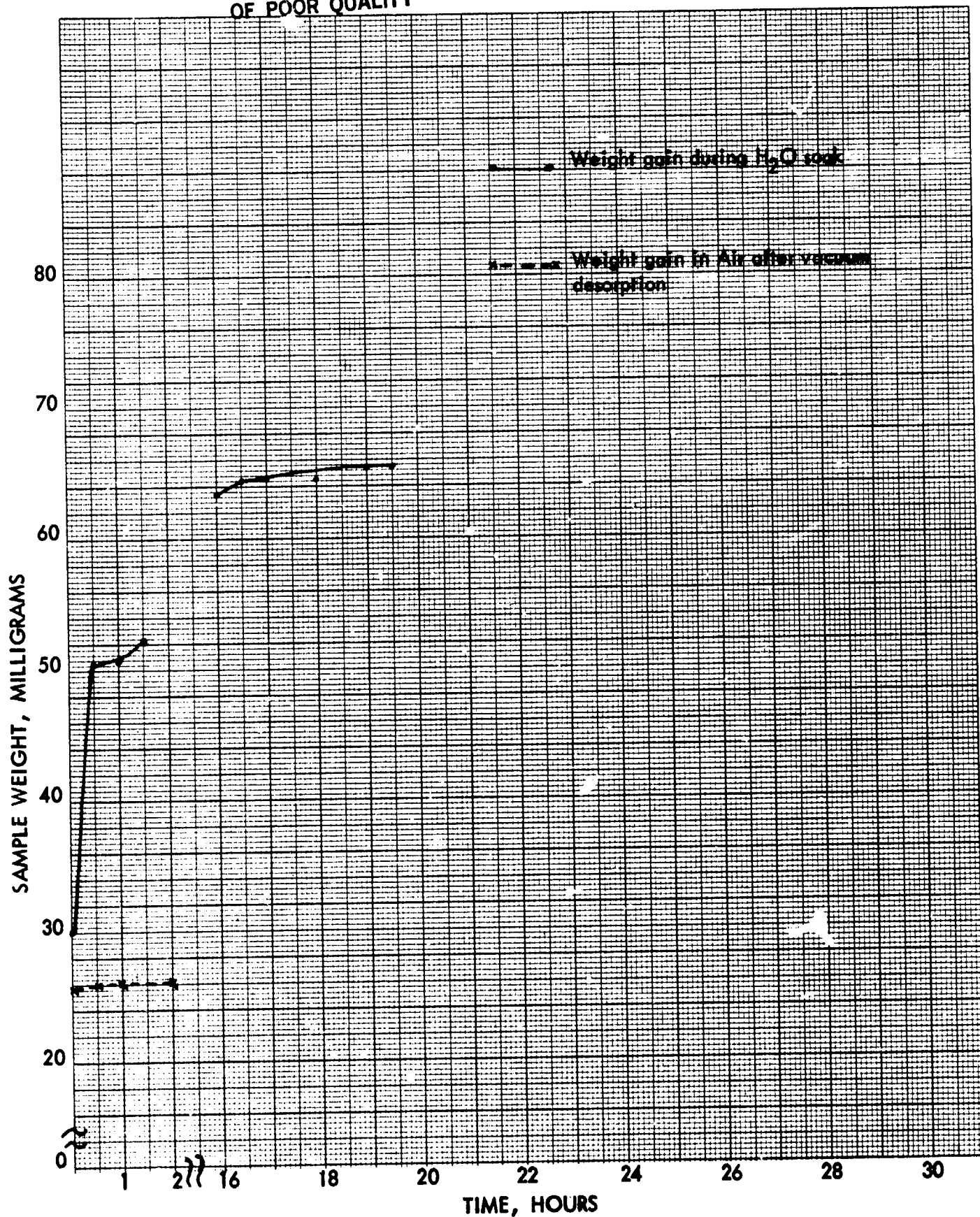


FIGURE 15. WEIGHT GAIN OF CORK DURING H₂O SOAK AND IN AIR AFTER VACUUM DESORPTION

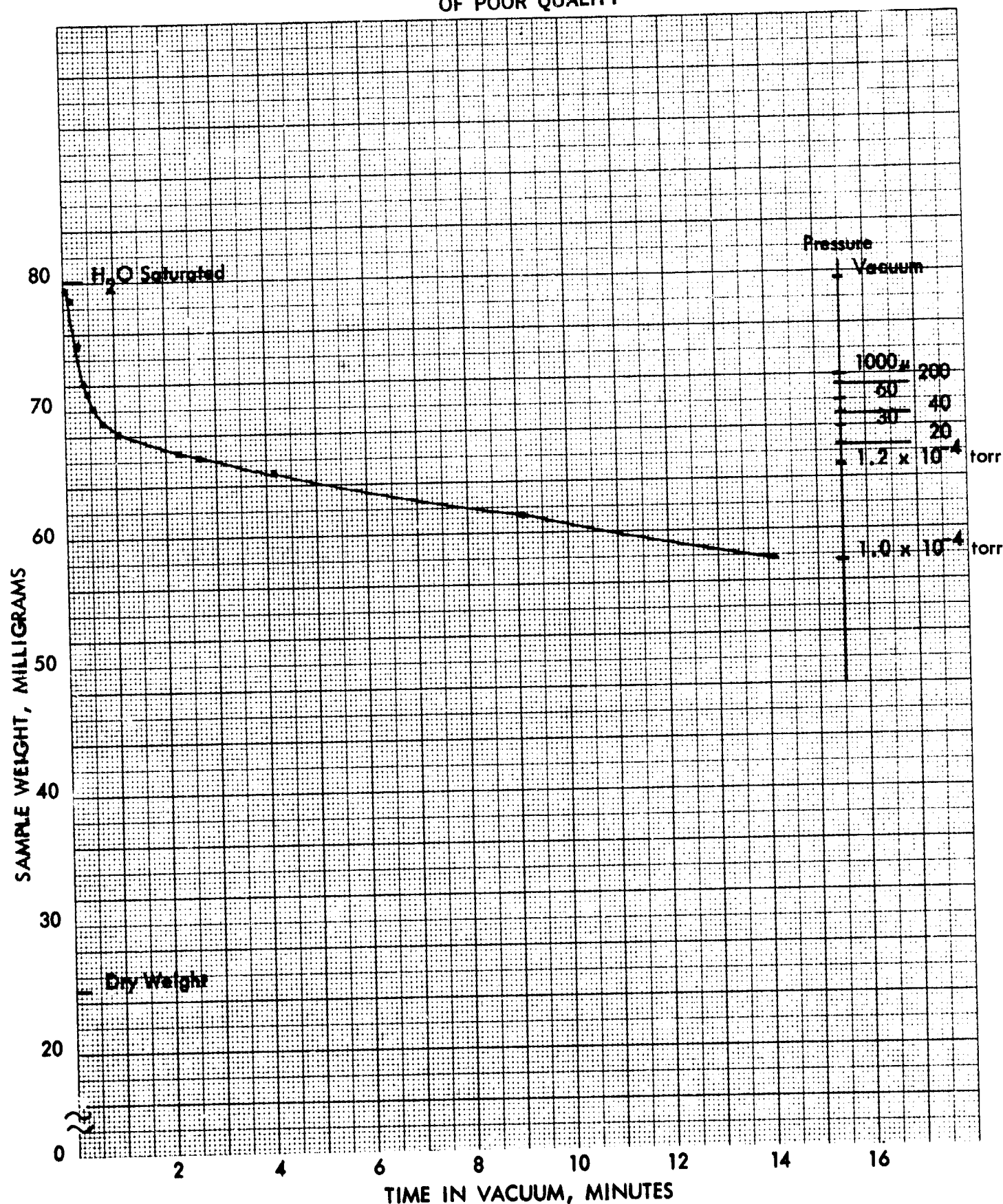


FIGURE 16. WATER DESORPTION OF MSA-1 IN VACUUM

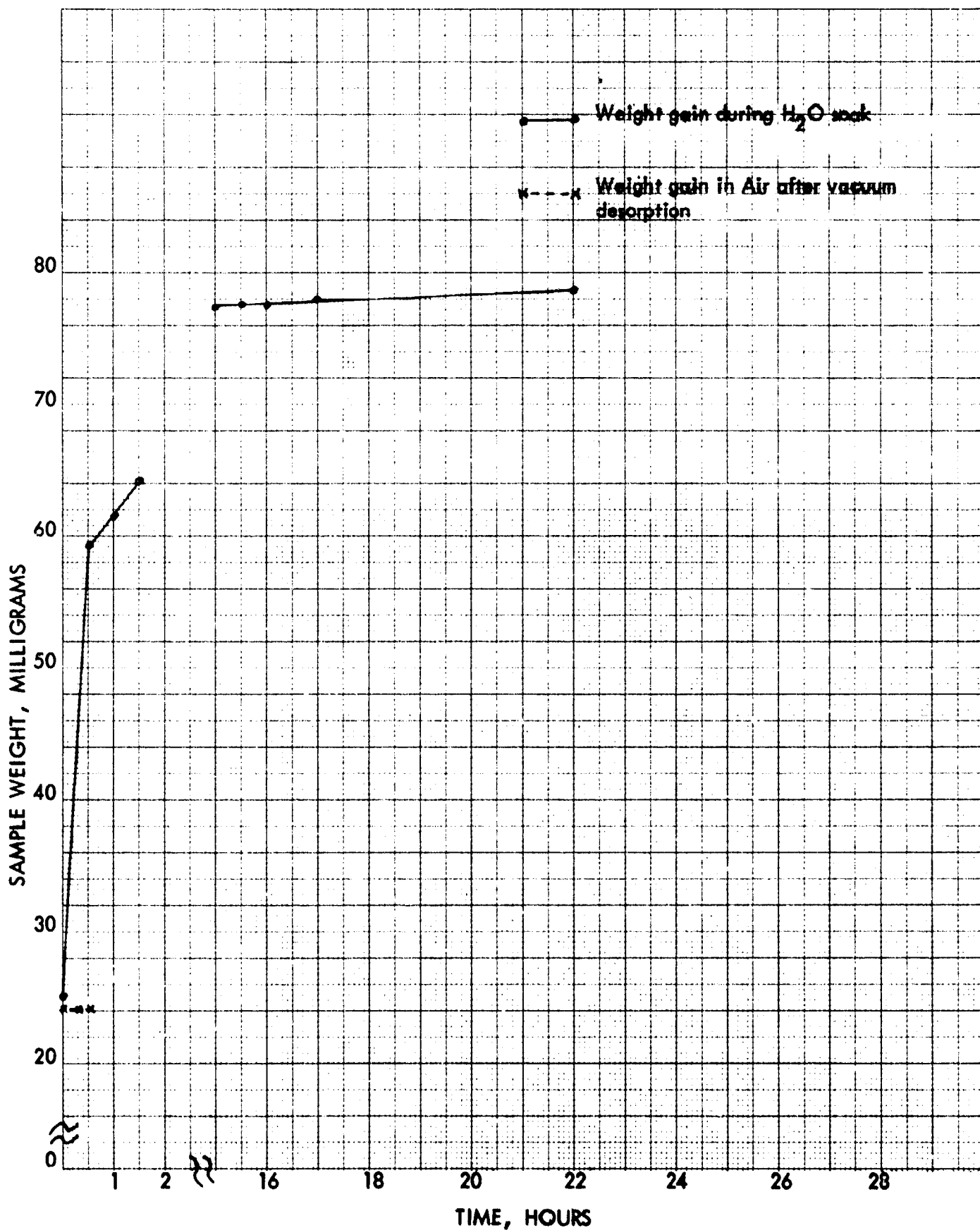


FIGURE 17. WEIGHT GAIN OF MSA-1 DURING H_2O SOAK AND IN AIR AFTER VACUUM DESORPTION

TABLE III. COMPARISON OF WATER ABSORPTION/DESORPTION CHARACTERISTICS OF SHUTTLE ABLATOR AND INSULATION MATERIALS

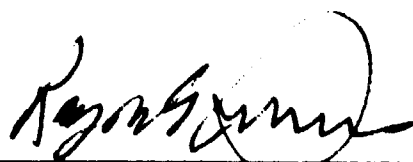
Material Characteristic	Orbiter Tile	488 Foam	SLA-561	Cork	MSA-1
Water Absorption in 24 Hrs (Large Volume/Surface Specimens)	+1.1% (2.5 cm Cube)	+40% (2.6 cm Cube)	+22% (2.6 cm Cube)	+60% (2.6 x 2.6 x .6) cm	+161% (2.6 x 2.6 x 2.6)cm
Water Absorption to Saturation Limit (Small Volume/Surface Specimens)	+14% (1.25 x .6 x .5) cm	+224% (2.1 x .65 x .5) cm	+62% (1 x .5 x .5) cm	+126% (.5 x .4 x .3) cm	+217% (.7 x .4 x .4) cm
Vacuum Desorption from Saturation - 1 Minute	Dry Weight	Dry Weight +75%	Dry Weight +73%	Dry Weight +81%	Dry Weight +78%
- 9 Minutes	Dry Weight	Dry Weight +15%	Dry Weight +36%	Dry Weight +70%	Dry Weight +67%
Rate of Absorption in Water	Slow	Moderate	Moderate	Rapid	Rapid
Absorption of Water in Ambient Laboratory Environment (35 - 45% Humidity) After Vacuum Desorption	Negligible	Negligible	Moderate	Moderate	Negligible

APPROVAL

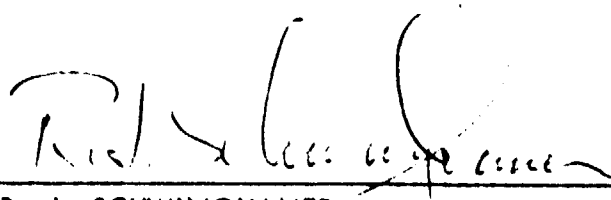
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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



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